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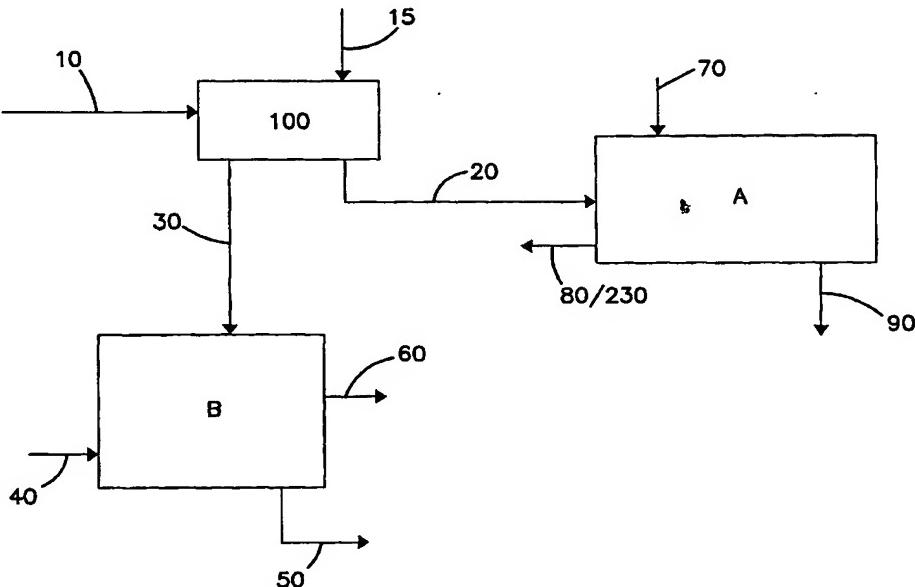
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(54) Title: SYSTEM FOR LIQUID EXTRACTION, AND METHODS



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(57) Abstract: A process for removing water from solid material (10) using liquid-solid extraction and liquid-liquid extraction in an extraction system (100). Multiple solvents are used sequentially to replace the water with a first solvent, then replacing that solvent with a second solvent, etc., then eventually removing the last solvent from the solid materials. The solvents have progressively lower heats of vaporization, enthalpy of vaporization, boiling point or related property, so as to conserve use of thermal energy.

## SYSTEM FOR LIQUID EXTRACTION, AND METHODS

This application is being filed as a PCT International Patent

Application in the name of Karges-Faulconbridge, Inc., a U.S. national corporation and resident, (Applicant for all countries except US) and Robert A. Wills a U.S. citizen and resident (Applicant for US only), on 19 December 2003, designating all countries and claiming priority to U.S. serial no. 60/435,015 filed 19 December 2002.

### Field of the Invention

The invention is directed to a system, and its methods of use, for extracting liquid water and/or a hydrocarbon from a feed stream using at least two solvents. The system and method can generally be described as a reduced energy extraction and drying processes.

### Background of the Invention

For many processes, an exiting stream, whether considered a waste stream, a by-product, or the main desired stream, is composed of a solid material wet with water. This water is typically found in both the interstitial spaces of the solid and is absorbed or adsorbed by the solid. Water such as this has typically been removed by drying the solids with thermal energy. This process generally requires a large amount of heat or energy to remove the water from the solids and obtain dry, usable solids.

Attempts have been made to use organic solvents to remove water from wet solids using solvents such as hexane. Essentially, the hexane is used to displace the water from the solids. The hexane remaining with the solids is then evaporated from the solids with thermal energy. Again, this process generally requires a large amount of thermal energy, but less than if water alone was being dried from the solids. However hexane also brings with it certain other concerns, such as toxicity. Further, because of poor displacement, large amounts of residual water may remain with the solids.

Some examples of known extraction methods include Baird, U.S. Patent No. 4,251,231, which utilizes liquid-liquid extraction to directly extract alcohol suitable for use in gasohol from a fermentation mixture. Gasoline was used

as the extraction solvent. The water was removed by either the use of adsorbents or absorbents, or by chilling the extracted alcohol-gasoline product to a temperature below about -10 °F, thereby removing the water.

During the ethanol manufacturing process, solids, wet with primarily  
5 water and some ethanol, exit the fermentation process as a beer stream. Other materials, such as oils and glycerol are also present in the beer stream. It is desired to obtain individual output streams of dry solids, water, and ethanol.

The beer stream solids, as discussed above, have the water in both the interstitial spaces of the solid and that which is absorbed or adsorbed by the solid.  
10 This water, and any ethanol, has typically been removed by drying the solids with thermal energy. Preferably, the ethanol is recovered and is used; unfortunately, recovery of pure, or fairly pure ethanol, is not usual. Additionally, preferably the water is sufficiently pure that the water can be readily disposed; unfortunately, the water has contaminants that inhibit direct, unmanaged disposal. Still further,  
15 contaminants, such as oils and glycerol, remain in the solids, making them undesirable for many applications.

What is needed is a low cost, more heat or energy efficient process for drying solids wet with water. It would be beneficial if the various output streams from the process could be reclaimed and used.

20

### Summary of the Invention

The invention is a process for separating water from solids and from other hydrocarbons that may be present, the process utilizing at least 20% less energy than conventional forced air drying of the same material.

Solids, wetted with water, are separated from the water and dried by  
25 the inventive process. The process removes the water residing in the interstitial spaces of the solids, as well as some of the water that has been absorbed by the solids. The process uses a liquid-solid extraction process to remove the water from the solid feed stream.

In one embodiment, multiple solvents are used to step-wise remove  
30 the water from the solids and obtain dry solids. The multiple solvents facilitate the removal of the water from the solids, by step-wise replacing the water with a solvent, replacing that solvent with a further solvent, and then eventually removing

the further solvent from the solids. Use of multiple solvents facilitates the separation of the initial solvent from the water and from the various solvents used in further processing. The multiple solvents are separated from each other by liquid-liquid extraction or distillation processes.

5           Multiple solvents utilize less thermal energy to dry the solids and separate the solvents than conventionally used in drying processes. The first solvent selected has a lower heat of vaporization, enthalpy of vaporization, boiling point, or other such physical property, than water. Each subsequent solvent has a still lower heat of vaporization, enthalpy of vaporization, boiling point, or other such physical 10          property than the previous solvent used.

In a further embodiment, the invention is directed to a process for drying solids initially wet with water. The process includes contacting a feed stream comprising solids having interstitial spaces, and water present in the interstitial spaces, with a first solvent. The water present in the interstitial spaces is displaced 15          by the first solvent, leaving the first solvent in the interstitial spaces. The feed stream having the first solvent in the interstitial spaces is then contacted with a second solvent; and the first solvent present in the interstitial spaces is displaced by the second solvent, thus providing the second solvent in the interstitial spaces.

20          In another embodiment, the process includes providing an ethanol source stream and an n-propyl bromide source stream. The water present in the interstitial spaces is displaced by the ethanol, leaving ethanol in the interstitial spaces. The ethanol in the interstitial spaces is then displaced by n-propyl bromide, leaving n-propyl bromide in the interstitial spaces. The n-propyl bromide is removed from the solids by the application of heat. In a preferred embodiment, an alcohol product that 25          is at least 90% pure ethanol is obtained.

25          In yet another embodiment, the process includes providing an ethanol source stream and an ether source stream. The water present in the interstitial spaces is displaced by the ethanol, leaving ethanol in the interstitial spaces. The ethanol in the interstitial spaces is then displaced by ether, leaving ether in the interstitial spaces. 30          The ether is removed from the solids by the application of heat. In a preferred embodiment, an ether product that is at least 95% pure ether is obtained. Alternately or additionally, an alcohol product that is at least 90% pure ethanol is obtained.

**Brief Description of the Drawings**

FIG. 1 is a schematic diagram of a general process according to the present invention, having an 'initial' separation subprocess, a 'solvent-from-solids' separation subprocess and a 'water-from-solvents' separation subprocess.

5 FIG. 2 is a schematic process diagram of a general, first embodiment of the 'introductory' separation subprocess according to the present invention.

FIG. 3A is a schematic diagram of an extraction unit of the subprocess of FIG. 2;

10 FIG. 3B is an enlarged, perspective view of a portion of the extraction unit of FIG. 3A;

FIG. 4 is a schematic process diagram of a first embodiment of the 'solvent-from-solids' separation subprocess according to the present invention;

FIG. 5 is a schematic process diagram of a portion of the 'solvent-from-solids' separation subprocess of FIG. 4.

15 FIG. 6 is a schematic process diagram of a first embodiment of the 'water-from-solvents' separation subprocess according to the present invention.

FIG. 7 is a schematic process diagram of a preferred process according to the present invention.

20 FIG. 8 is a schematic diagram of an alternate process according to the present invention.

FIG. 9 is a schematic diagram of another alternate process according to the present invention.

FIG. 10 is a schematic diagram of yet another alternate process according to the present invention.

25 FIG. 11 is a binary diagram for a preferred three-solvent system according to the present invention.

**Detailed Description of the Preferred Embodiment**

As provided above, the invention is directed to processes for separating water from solids by utilizing at least two solvents. The process uses a 30 first solvent to displace the water from the interstitial spaces in the solids. This first solvent, having a lower heat of vaporization and boiling point than the water, is easier to remove from the solids than water. A second solvent is used to displace the

first solvent from the solids. The second solvent has a lower heat of vaporization and boiling point than the first solvent.

The first solvent is preferably soluble in water but preferably does not form an azeotropic mixture with water. An azeotropic mixture is a mixture of two 5 or more substances that behaves like a single substance. The vapor produced by partial evaporation of the liquid has the same composition as the liquid; that is, vaporization of the mixture does not result in separation of the initial substances.

The second solvent is preferably soluble with the first solvent but insoluble with water. Additionally, the first and second solvents should preferably 10 not form an azeotropic mixture.

Any third, or subsequent solvent, is preferably soluble with the predecessor solvent, but not form an azeotropic mixture with the predecessor solvent.

By utilizing this multiple solvent, liquid-extraction process, the 15 energy needed to dry the solids and to separate the various solvents from each other and from water, is greatly reduced compared to conventional processes.

The processes of the invention can generally be reduced to an initial extraction subprocess that removes water from the solids followed by two subprocesses, a 'solvent-from-solids' separation subprocess which separates solvent 20 from the solids, and a 'water-from-solvents' separation subprocess that separates and reclaims the water and solvents, and optionally, other components.

Referring now to the figures, a diagrammatic rendering of the process according to the present invention is generally depicted in FIG. 1. This process has an initial separation process 100 to separate water from the solids and two general 25 subprocesses, one for removing solvent from solids, subprocess A, and a second for separating and reclaiming water and solvents, subprocess B.

Feed stream 10, an aqueous stream with solids therein, is illustrated entering the system at the top left corner of FIG. 1. The type and amount of solids in stream 10 will vary. The specific solids present will depend on the source, and 30 example sources include grains, other plant materials and earthen materials.

The amount of solids in stream 10 is generally 5 to 50% by weight. A common amount of solids in stream 10 is about 10-12%. As mentioned, steam 10 is typically an aqueous stream, with the water present at a level of about generally

50 to 95% by weight. A common amount of water in stream 10 is about 78 wt-%. Other liquids, in addition to the water, can be and are often present in feed stream 10.

If feed stream 10 is from a fermentation process, stream 10 generally 5 includes alcohol (such as ethanol). The level of alcohol and other components in stream 10 is dependent on the efficiency of the process providing stream 10, however, the alcohol in stream 10 is generally less than 16 wt-%. Usually, the level of alcohol in stream 10 is greater than about 8 wt-%. A common level of alcohol in some streams is about 15 wt-%.

10 Other materials are typically present in stream 10. For example, oil (such as corn oil) and glycerol are usually present. Examples of solutes that may be present include acids (such as acetic acid), aldehydes (such as acetaldehyde), and various sugars. The levels of these material are low, typically less than 2 wt-% and often less than 1 wt-% of stream 10.

15 Returning to FIG. 1, feed stream 10 is fed into a water/solid extraction system 100 where the solids of feed stream 10 are separated from water. An alternate descriptive term for water/solid extraction system 100 is a water extractor or a solid-liquid extraction unit. Extraction system 100 is configured to remove water from feed stream 10 and replace the water with a solvent. Additional 20 details regarding a preferred extraction system 100 are provided below.

Extraction system 100 transfers one or more components from feed stream 10 into the extraction solvent stream (described below). Typically, extraction system 100 operates in a counter-current arrangement; that is, the extraction solvent stream enters system 100 farthest from where feed stream 10 enters, and the two 25 streams contact and pass counter-currently to each other.

In addition to feed stream 10 being fed into extraction system 100, an extraction solvent stream 15 is fed into system 100. It is the solvent in stream 15 that will extract and replace the water from feed stream 10. First solvent from stream 15 combines with or displaces the original aqueous liquids from feed stream 30 10 as feed stream 10 and solvent stream 15 pass in system 100.

This exchange of one solvent for another in a stream is due to concentration equilibrium. Solvent, present at a high concentration in stream 15, moves to a stream having a lower concentration, i.e., stream 10; likewise, water,

present at a high concentration in stream 10, moves to a stream with a lower concentration of water, i.e., stream 15.

The solvent is selected for stream 15 based on a lower heat of vaporization or enthalpy of vaporization than the water in feed stream 10. Water has 5 a heat of vaporization of 1000 BTU per pound of water, thus, solvent of stream 15 should have a heat of vaporization less than 1000. The lower the heat of vaporization in relation to 1000 BTU, the easier the subsequent separation of solvent 15 from water. Preferably, the solvent of stream 15 is water soluble, however, it is preferred that the solvent of stream 15 and water do not form an azeotropic mixture, 10 so that subsequent separation of the solvent and water is simple.

Although virtually any water soluble solvent can be selected for extraction solvent stream 15, it is preferred to select one which may already be present in feed stream 10. Examples of suitable solvents include alcohols (such as ethanol, methanol, isopropyl alcohol, and gasohol) and ketones (such as acetone, 15 methyl ethyl ketone (MEK), methyl isobutyl ketone (MIBK)). If feed stream 10 is primarily solids, water and alcohol, as it is from many fermentation processes, solvent stream 15 is preferably an alcohol, more preferably the alcohol that is present in feed stream 10.

As stated above, water in feed stream 10 is replaced with first solvent 20 from stream 15 by water extraction system 100. The resulting output streams from system 100 are solids stream 20 and liquid stream 30.

Solid stream 20 is a wet solids stream, composed of the solids from stream 10 and an amount of first solvent from stream 15. Wet solids stream 20 progresses to and is treated by subprocess A, as will be described below. Liquid 25 stream 30 is generally composed of the original liquid from feed stream 10 (that is, the water and any other liquid, such as an alcohol) and the solvent from solvent stream 15. Liquid stream 30 progresses to and is treated by subprocess B, described below.

30

### Extraction System 100

A preferred configuration for a water-solid extraction system 100 is illustrated in FIG. 2. As seen in FIG. 1 and in FIG. 2, feed stream 10 and solvent

stream 15 enter system 100, and wet solids stream 20 and liquid stream 30 exit system 100.

Water-solid extraction system 100 has at least one extraction unit 110. In the system 100 illustrated in FIG. 2, system 100 has three extraction units, 5 specifically, 110A, 110B, 110C. Each extraction unit 110 includes a mixing tank 112, a pump 114, mechanical separator 116, and the piping to operably connect the elements.

Mixing tank 112 can be any suitable receptacle for combining and temporarily storing solid and liquid materials. In the embodiment illustrated, tank 10 112 accepts beer feed 10 and water/solvent stream 31, which will be described below. Examples of suitable materials for tank 112 include steels, such as carbon steel and stainless steels. A preferred material is 304 stainless steel. The volume of tank 112 is based on the material flow volumes and desired residence time in tank 112. A 30 gallon tank is a suitable size for some processes.

15 Pump 114, used to move material from tank 112, is positioned downstream of tank 112. Pump 114 is selected for its ability to move the material from tank 112, which includes solid material and liquid, to mechanical separator 116. Examples of suitable pumps include diaphragm pumps, centrifugal pumps, and pumps designed to pump a combination of liquid and solids. An example of a 20 preferred pump 114 is a centrifugal pump available from Goulds Pumps of ITT Industries.

Mechanical separator 116 separates solid material from liquid. Examples of suitable mechanism separating equipment include Rotocel extractors, double screw extractors, baskets, rotary perforated belts, sliding rolls, and loop 25 extractors; this equipment is well known for solid/liquid extraction processes. The specific equipment used will be dependent on the solvents used in the process and in the solvent ratios. Preferred equipment for use in extraction system 100 is a stationary screen, described below.

The piping connecting tank 112, pump 114, and mechanical separator 30 116, for each extraction unit 110, is selected for its ability to move the solid-liquid material. An example of preferred piping is 1 inch carbon steel piping.

A preferred configuration for a screen mechanical separator 116 is illustrated in FIG. 3A. Separator 116 has a housing 1162 in which is a screen 1163.

Screen 1163 has a first, curved portion 1163A and a second, generally vertical portion 1163B. Screen 1163 separates housing 1162 into a filtrate side 1167 and a cake side 1168.

A nozzle 1164 is present to spray wet solids stream 11, from tank 5 112, onto screen 1163. In one preferred process configuration, nozzle 1164 is configured to provide a flow of 8-10 gallons/minute of wet solids stream 11 onto screen 1163.

An enlargement of screen 1163 is illustrated in FIG. 3B. Screen 1163 has a plurality of separating members 1165 secured by cross-members 1166, both of 10 which can be carbon steel or stainless steel. Separating members 1165, positioned closer to nozzle 1164, on cake side 1168, preferably extend vertically, to facilitate solids running down members 1165. In one preferred process, members 1165 and 1166 are arranged to provide a mesh size (i.e., an opening) of at least 0.01 inch.

Wet solids stream 11, sprayed by nozzle 1164 primarily onto curved 15 portion 1163A, is separated by members 1165 and 1166. Liquid from stream 11 passes through screen 1163 and is collected on filtrate side 1167. The solids, too large to pass through screen 1163, remain on cake side 1168.

It is understood that some liquid will not pass through screen 1163 but will remain with the solids. Screen 1163 may have a dam or baffle 1169 20 positioned at or near the juncture of curved portion 1163A and vertical portion 1163B, to retain solids in an attempt to have liquid drop therefrom.

The liquid, having passed through screen 1163 to filtrate side 1167, would be removed from housing 1162 via an outlet 1167A. The wet solids, left on cake side 1168, would be removed from housing 1162 via an outlet 1168A.

25 Returning to FIG. 2, the illustrated process has three extraction units 110A, 110B, 110C. Unit 110A has mixing tank 112A, pump 114A, mechanical separator 116A, and the piping to operably connect the elements. Unit 110B has mixing tank 112B, pump 114B, mechanical separator 116B, and the piping to operably connect the elements. Unit 110C has mixing tank 112C, pump 114C, 30 mechanical separator 116C, and the piping to operably connect the elements.

Beer feed 10 is fed into tank 112A where it is mixed with water/solvent stream 31 (described later). This mixture, as stream 11, is pumped via

pump 114A to mechanical separator 116A, where it is split into water/solvent stream 30 and wet solids stream 34.

5 Wet solids stream 34 is fed into tank 112B where it is mixed with water/solvent stream 32 (described later). This mixture, as stream 12, is pumped via pump 114B to mechanical separator 116B, where it is split into water/solvent stream 31 and wet solids stream 35.

10 Wet solids stream 35 is fed into tank 112C where it is mixed with first solvent stream 15. This mixture, as stream 13, is pumped via pump 114C to mechanical separator 116C, where it is split into water/solvent stream 32 and wet solids stream 20.

15 Stream 30, from unit 110A, is referred to as a "full miscella". In the embodiment illustrated in FIG. 2, because there are three units, each stream is allotted a third (i.e., 1/3) designation. Stream 31, from unit 110B, is referred to as a "2/3 miscella" and stream 32, from unit 110C, is referred to as a "1/3 miscella". Full miscella stream 30 has a lower solvent concentration and a higher water concentration than 2/3 miscella stream 31, which has a lower solvent concentration and a higher water concentration than 1/3 miscella stream 32.

20 Each of these stream 30, 31, 32 is reused in the process. Stream 31 is recycled and fed into tank 112A, and stream 32 is recycled and fed into tank 112B. Full miscella stream 30, composed of water from beer feed 10 and first solvent from stream 15, is used in 'water-from-solvents' separation subprocess B. Wet solids stream 20, composed of solids and first solvent from stream 15, progresses to 'solvent-from-solids' separation subprocess A.

25 **'Solvent-from-Solids' Separation Subprocess A**

Returning to FIG. 1, from water extraction system 100, wet solids stream 20 is conveyed to 'solvent-from-solids' separation subprocess A. In subprocess A, solvent from wet solids stream 20 is removed, by using a second solvent, to obtain dry solid stream 90. Second solvent is introduced to subprocess A as stream 70. First solvent (originally from stream 15) and second solvent from stream 70 depart subprocess A as stream 80/230.

In subprocess A, the first solvent from stream 15, such as an alcohol, is extracted from the solids and replaced with a second solvent. The second solvent

is removed from the solids and dry solids are obtained. 'Solvent-from-solids' separation subprocess A is generally configured as two sub-subprocess, solvent extraction and thermal drying.

Referring to FIG. 4, 'solvent-from-solids' separation subprocess A is illustrated having solvent extraction system 200 and drying system 300. An alternate descriptive term for solvent extraction system 200 is a solvent extractor or a solid-liquid or solid-solvent extraction unit. Solvent extraction system 200 is configured to remove the first solvent from wet solids stream 20 and replace the first solvent with a second solvent.

Solvent extraction system 200 transfers one or more components from wet solids stream 20 into the extraction second solvent stream (described below). Typically, solvent extraction system 200 operates in a counter-current arrangement.

In addition to wet solids stream 20 being fed into extraction system 200, an extraction second solvent stream 70 is fed into system 200. It is the solvent in stream 70 that will extract and replace the solvent from wet solids 20. Second solvent from stream 70 combines with or displaces the first solvent from feed stream 15 in solids stream 20 as stream 20 and solvent stream 70 pass in system 200.

The second solvent is selected for stream 70 based on a lower heat of vaporization or enthalpy of vaporization than the first solvent of stream 15, which is present in wet solids stream 20. Preferably, the solvent of stream 70 is soluble with and miscible with the first solvent of stream 15, however, it is preferred that the solvent of stream 70 and the solvent of stream 15 do not form an azeotropic mixture, so that subsequent separation of the solvents is simple.

Examples of suitable solvents for stream 70 include ethers, (such as ethyl ether, MTBE (methyl tert-butyl ether), ETBE (ethyl tert-butyl ether), fluorinated ethers, and other low molecular weight ethers), halogenated hydrocarbons (n-propyl bromide or 1-bromopropane, commercially available under the trade name "Hypersolve NPB"), straight chain low molecular hydrocarbons (such as hexane, pentane), and low molecular weight aromatic hydrocarbons (such as toluene, benzenes, xylenes).

The second solvent is selected on the basis of high solubility with the first solvent (e.g., ethanol), low solubility with water, and ease of separation between

the first and second solvents, generally based on differential of heat of vaporization or enthalpy of vaporization.

Stream 70 may be provided by an external source, but is preferably recycled from the solvent removed from the solids, and from overhead stream 70  
5 from still 700, as will be discussed below.

As stated above, first solvent from stream 15, now present in wet  
solids stream 15, is replaced with second solvent from stream 70 by solvent  
extraction system 200. The resulting output streams from system 200 are wet solids  
10 stream 220 and liquid stream 230; see FIG. 4. Solid stream 220 is composed of the  
solids and an amount of second solvent from stream 70. Wet solids stream 220  
progresses to drying system 300, where the second solvent is removed from the  
solidid.  
15

Liquid stream 230 is generally composed of the solvent from solvent  
stream 15 and second solvent from stream 70. Liquid stream 230 progresses to and  
is treated by subprocess B, described below.

#### Solvent Extraction System 200

A preferred configuration for a solvent-solid extraction system 200 is  
illustrated in FIG. 5. As seen in FIG. 4 and in FIG. 5, wet solids stream 20 and  
20 second solvent stream 70 enter system 200, and wet solids stream 220 and liquid  
stream 230 exit system 200.

Solvent-solid extraction system 200 has at least one extraction unit  
210. In the system 200 illustrated in FIG. 5, system 210 has three extraction units,  
specifically, 210A, 210B, 210C. Each extraction unit 210 includes a mixing tank  
25 212, a pump 214, mechanical separator 216, and the piping to operably connect the  
elements.

Mixing tank 212 can be any suitable receptacle for combining and  
temporarily storing solid and liquid materials. In the embodiment illustrated, tank  
212 accepts wet solids stream 20 and liquid stream 41, which will be described  
30 below. Examples of suitable materials for tank 212 include steels, such as carbon  
steel and stainless steels. A preferred material is 304 stainless steel. The volume of  
tank 212 is based on the material flow volumes and desired residence time in tank  
212. A 30 gallon tank is a suitable size for some processes.

Pump 214, used to move material from tank 212, is positioned downstream of tank 212. Pump 214 is selected for its ability to move the material from tank 212, which includes solid material and liquid, to mechanical separator 216. Examples of suitable pumps include diaphragm pumps, centrifugal pumps, and 5 pumps designed to pump a combination of liquid and solids. An example of a preferred pump 214 is a centrifugal pump available from Goulds Pumps of ITT Industries.

Mechanical separator 216 separates solid material from liquid. Examples of suitable mechanism separating equipment include Rotocel extractors, 10 double screw extractors, baskets, rotary perforated belts, sliding rolls, and loop extractors; this equipment is well known for solid/liquid extraction processes. The specific equipment used will be dependent on the solvents used in the process and in the solvent ratios. Preferred equipment for use in extraction system 200 is a stationary screen, described below.

15 The piping connecting tank 212, pump 214, and mechanical separator 216, for each extraction unit 210, is selected for its ability to move the solid-liquid material. An example of preferred piping is 1 inch carbon steel piping.

20 A preferred configuration for a screen mechanical separator 216 is illustrated in FIG. 3A as separator 116; that is, mechanical separator 216 can be the same as mechanical separator 116 from water extraction system 100.

25 Returning to FIG. 5, the illustrated process has three extraction units 210A, 210B, 210C. Unit 210A has mixing tank 212A, pump 214A, mechanical separator 216A, and the piping to operably connect the elements. Unit 210B has mixing tank 212B, pump 214B, mechanical separator 216B, and the piping to operably connect the elements. Unit 210C has mixing tank 212C, pump 214C, 30 mechanical separator 216C, and the piping to operably connect the elements.

Wet solids stream 20 is fed into tank 212A where it is mixed with liquid stream 41 (described later). This mixture, as stream 21, is pumped via pump 214A to mechanical separator 216A, where it is split into liquid stream 230 and wet solids stream 44.

Wet solids stream 44 is fed into tank 212B where it is mixed with liquid stream 42 (described later). This mixture, as stream 22, is pumped via pump

214B to mechanical separator 216B, where it is split into liquid stream 41 and wet solids stream 45.

5 Wet solids stream 45 is fed into tank 212C where it is mixed with second solvent stream 70. This mixture, as stream 23, is pumped via pump 214C to mechanical separator 216C, where it is split into liquid stream 42 and wet solids stream 220.

Liquid stream 230, from unit 210A, is referred to as a "full miscella". Stream 41, from unit 210B, is referred to as a "2/3 miscella" and stream 42, from unit 210C, is referred to as a "1/3 miscella". Full miscella stream 230 has a lower 10 second solvent concentration and a higher first solvent concentration than 2/3 miscella stream 41, which has a lower second solvent concentration and a higher first solvent concentration than 1/3 miscella stream 42.

Each of these streams 230, 41, 42 is reused in the process. Stream 41 is recycled and fed into tank 212A, and stream 42 is recycled and fed into tank 15 212B. Full miscella stream 230, composed of first solvent from stream 15 and second solvent from stream 70, is used in 'water-from-solvents' separation subprocess B. Wet solids stream 220, composed of solids and second solvent from stream 70, progresses to drying system 300.

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### Drying System 300

Wet solids stream 220, having solids and second solvent from stream 70, from solvent extraction system 200, is fed to drying system 300, where the solvent and any other volatile liquids or solvents are removed from the solids. Drying system 300 is the only unit in 'solvent-from-solids' separation subprocess A 25 that uses thermal energy. Examples of suitable equipment for drying system 300 include a steam-jacketed tube dryer (such as a Schnecken tube dryer), steam-heated-screw tube dryer, a rotary dryer, a belt dryer, a down-draft desolventizer, or a DT; this equipment is well known for drying processes. A preferred drying system 300 includes a steam-jacketed tube style dryer.

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The solvent is thermally removed from the solids at drying system 300, and dry solids are obtained as output stream 90. The second solvent removed exits drying system 300 as stream 80. Stream 80 may be further processed. In the

process embodiment illustrated in FIG. 4, stream 80 is combined with miscella stream 230 and sent to 'water-from-solvents' separation subprocess B.

#### **'Water-from-Solvents' Separation Subprocess B**

5        Returning to FIG. 1, stream 30, composed of water from beer feed 10 and first solvent from stream 15, is conveyed to 'water-from-solvents' separation subprocess B and processed to separate the water from solvent.

10      However, to maximize the separation to provide desired output streams, subprocess B preferably utilizes a second solvent, provided to subprocess B as solvent stream 40.

15      Solvent of stream 40 is selected to have a lower heat of vaporization or enthalpy of vaporization than the components of stream 30, that is, the water from feed stream 10 and the solvent of stream 15. In a preferred method, the solvent of stream 40 is the same as the solvent of stream 70, from 'solvent-from-solids' separation subprocess A, described above. Preferably, solvent stream 40 is recycled from 'solvent-from-solids' separation subprocess B; specifically, solvent stream 40 is obtained from stream 80.

20      Stream 80 is combined with stream 230 and this combined stream 80/230 is fed as a single stream to subprocess B. Stream 40 is added as necessary to assure a proper concentration of the three major components, water, first solvent and second solvent.

25      Any known methods can be used to separate the water from the solvent. Examples of suitable liquid-liquid extraction or liquid-liquid separation methods include distillation, for example packed, York-Scheibel, Oldshue-Ruston, rotating disc, Karr or pulsed columns. Another suitable separation method is with a centrifugal contactor.

One general configuration for 'water-from-solvents' separation process B is illustrated in FIG. 6. Subprocess B includes a liquid-liquid separation unit 400 and two distillation units 500, 600.

30      In this embodiment, liquid stream 30, which enters liquid-liquid process unit 400 at the bottom, has a density less than stream 80/230 which enters at the top of unit 400. Thus the components of stream 30 rise in unit 400 while components in stream 80/230 fall in the column. Exiting from unit 400 are top

stream 45 from the top of unit 400 and a bottom stream 65 from the bottom of unit 400. The particular composition of streams 45, 65 will depend on the composition of streams 30 and 80/230. Stream 40 is a make-up stream to assure proper balance of water, first solvent and second solvent in unit 400.

5 There are components in each entering stream 30, 40 that are soluble in one another and some that are insoluble in each other. By choice, the solvent of stream 80/230 and water are typically not soluble in each other and form an upper and lower phase rich in one or the other. As the solvent may have a density greater or lesser than that of water, the water rich phase may be at the top or bottom. If the  
10 solvent of stream 80/230 is assumed to have a density of 1.3, and therefore denser than water, the solvent rich phase will exit out the bottom of the column 400 as stream 65 and the water-rich phase out the top as stream 45. Stream 45 tends to be a stream high in alcohol and water with other lesser water-soluble components, possibly with a small amount of the solvent of stream 80/230. Stream 65 is a stream  
15 high in solvent, with possibly small amounts of alcohol and other components.

Stream 45 is sent to process unit 500, an evaporation or distillation device, for further separation into streams 55 and 60. Stream 65 is sent to process unit 600, a different distillation or evaporation device, for further separation into streams 50 and 75.

20 In many processes, streams 50, 55, 60, 75 are sufficiently pure so that the material from these streams can be sold or otherwise used without the need for additional processing.

#### A Preferred Embodiment of the Process

A preferred embodiment of the process is diagrammatically  
25 illustrated in FIG. 7. This process has an initial extraction process that removes the water from the solids followed by two subprocesses. The first subprocess removes the initial solvent from the solids and a second subprocess that separates and reclaims the water, solvent and other components. The description of this preferred process uses the same reference numerals used before for like streams and  
30 equipment, as appropriate, except that the reference numerals are followed by an "a".

In this embodiment of a preferred process, a beer stream 10a (composed of corn solids, water, ethanol, oils, glycerol and other minor components) is fed into a solid-liquid extraction system 100a. An alternative term for solid-liquid extraction system 100a is a water extractor or water extraction unit. Water extraction system 100a is designed to remove water from the feed stream 10a and replace the water with a solvent. Examples of suitable solids-liquid extraction equipment have been described previously as water extraction system 100, and a preferred system 100a includes three separators 116. The water-extraction system 100a operates in a counter-current fashion.

5           A first solvent, an extraction solvent, 15a is fed into water extraction system 100a where part of the solvent replaces the water from stream 10a. In this embodiment, the extraction solvent is ethanol. Ethanol has a lower heat of vaporization or enthalpy of vaporization than the water in feed stream 10a.

10           System 100a, the resulting output streams are wet solids stream 20a and liquid stream 30a. Solid stream 20a progresses to and is treated by 'solvent-from-solids' separation subprocess A, described below. Liquid stream 30a progresses to and is treated by 'water-from-solvents' separation subprocess B, also described below.

20           Subprocess A

Wet solids stream 20a from system 100a is pumped to solids-liquid extractor system 200a by piping. Examples of suitable equipment for system 200a have been provided previously as solids-liquid extractor system 200, and a preferred system 200a includes three separators 216. Typically solvent extraction system 200a operates in a counter-current arrangement.

25           Also entering solvent extraction system 200a is a second solvent, stream 70a. In this embodiment, the solvent is n-propyl bromide. n-propyl bromide has a lower heat of vaporization or enthalpy of vaporization than the water in feed stream 10a and the ethanol of stream 15a.

30           In solvent extraction system 200a, ethanol in stream 20a, particularly that in the interstitial spaces of the solids, is replaced with n-propyl bromide from stream 70a. The ethanol leaves system 200a leave as stream 230a and the solids, now wet with n-propyl bromide exit system 200a as stream 220a.

Stream 220a is fed to a dryer 300a where n-propyl bromide and any other remaining volatile liquids or solvents are removed from the solids. Dryer 300a is the only unit in subprocess A that uses thermal energy. Examples of suitable equipment for dryer 300a have been previously described in respect to dryer 300.

- 5 Dry solids exit as output stream 90a. The thermally removed solvent exits dryer 300a as stream 80a, a vapor. Stream 80a is combined with liquid stream 230a. This combined stream 80a/230a and stream 30a is fed into liquid-liquid extraction unit 400a in 'water-from-solvents' separation subprocess B.

10

#### Subprocess B

- Combined stream 30a is provided to the bottom of process unit 400a. A solvent stream 80a/230a enters at the top of unit 400a. In this embodiment, the solvent of stream 80a/230a is n-propyl bromide. Stream 30a has a density less than n-propyl bromide, which enters at the top of unit 400a. Thus the components of stream 80a/230a fall in the column while components in stream 30a rise in the column. Normal-propyl bromide, with a density of 1.3, will therefore exit out the bottom of the column as a solvent-rich stream 65a, and the water-rich phase will exit out the top as stream 45a.

- 20 Stream 45a is high in alcohol and water content with other lesser water-soluble components. There may be a small amount of n-propyl bromide in stream 45a.

- 25 Stream 45a is sent to process unit 500a, an evaporation or distillation device. Unit 500a separates the ethanol from the mixture of stream 45a; the ethanol, as a vapor and as an azeotrope of ethanol and water, leaves unit 500a as stream 60a. Stream 60a may either be condensed, used as is, or sent for further processing to remove other components. Stream 60a may also be conveyed, as a vapor, to other purification devices to provide a product ethanol that is 99.9+% pure.

- 30 Stream 55a from process unit 500a is mostly water with some water soluble components that did not vaporize in unit 500a. This liquid stream 55a may be used as is or further refined or purified.

Returning to unit 400a, stream 65a, the high organic bottom stream from unit 400a, is also sent to a distillation or evaporation device. The majority of stream 65a consists of n-propyl bromide and the remainder of stream 65a is

composed of fat soluble components, such as corn oil. Stream 65a feeds process device 600a which has an exiting vapor stream 75a and a liquid stream 50a. Stream 75a is primarily n-propyl bromide. This vapor can be condensed and recycled (reused) in the solid-liquid extraction subprocess A, as stream 70a. The liquid stream 50a is primarily fats and oils; this stream may be used as is or may be further refined.

#### Alternate Embodiments of the Process

A first alternate embodiment of the process is diagrammatically illustrated in FIG. 8. This process has an initial extraction process that removes the water from the solids followed by two subprocesses. The first subprocess removes the initial solvent from the solids and a second subprocess that separates and reclaims the water, solvent and other components. The description of this preferred process uses the same reference numerals used before for like streams and equipment, as appropriate, except that the reference numerals are followed by a "b".

In this embodiment, feed stream 10b is fed into a solid-liquid extraction system 100b where the solids of feed stream 10b are separated from the water. Examples of suitable solids-liquid extraction equipment have been described previously as water extraction system 100, and a preferred system 100b includes separators 116.

An extraction solvent stream 15b is fed into water extraction system 100b with feed stream 10b. In this embodiment, the extraction solvent is ethanol. Ethanol has a lower heat of vaporization or enthalpy of vaporization than the water in feed stream 10b. The resulting output streams from system 100b are wet solids stream 20b and liquid stream 30b. Solid stream 20b progresses to and is treated by 'solvent-from-solids' separation subprocess A, described below. Liquid stream 30b progresses to and is treated by 'water-from-solvents' separation subprocess B, also described below.

#### Subprocess A

From water extraction system 100b, solid stream 20b is conveyed to solid-liquid extraction system 200b where the solvent from stream 15b is removed from the solids and replaced with second solvent entering as stream 70b. In this

embodiment, the solvent is ethyl ether, which has a lower heat of vaporization or enthalpy of vaporization than the water in feed stream 10b and the ethanol of stream 15b. The ethyl ether of stream 70b may be provided by an external source, but is preferably recycled from the solvent removed from the solids, and from overhead stream 75b from still 600b, as will be discussed below.

In solvent extraction system 200b, ethanol in stream 20b is replaced with ethyl ether from stream 70b. The ethanol leaves system 200b as stream 230b and the solids, now wet with ethyl ether, exit system 200b as stream 220b.

Stream 220b is fed to a dryer 300b where ethyl ether and any other 10 remaining volatile liquids or solvents are removed from the solids. The thermally removed solvent exits dryer 300b as stream 80b, a vapor, and progresses to condenser 800. Depending on the volume of stream 80b, a portion of it may be removed as an ether side-stream. The remainder of stream 80b is returned to system 200b.

15 Stream 230b progresses to 'water-from-solvents' separation subprocess B.

#### Subprocess B

'Water-from-solvents' separation subprocess B treats liquid stream 30b from water extraction system 100b and stream 230b from subprocess A. Stream 20 30b is provided to the top of process unit 400b. Liquid-liquid extraction unit 400b is typically a tall column with four ports, one inlet at the top and one inlet at the bottom, and two outlets, one at the top and one at the bottom; streams from the two inlets run counter-current. A solvent stream 40b enters at the bottom of unit 400b. In this embodiment, the solvent of stream 40b is ethyl ether. Thus the components 25 of stream 40b rise in the column while components in stream 30b fall in unit 400b, resulting in exiting aqueous bottom exit stream 45b, which has a lower concentration of ethanol than stream 30b did at the inlet, having transferred some ethanol to the ether stream. Also exiting is top exit stream 65b, mostly ether but which has a higher concentration of ethanol than stream 40b did at the inlet, having received 30 some ethanol from stream 30b.

Bottom exit stream 45b is composed of the water, ethanol, and some other hydrocarbons from feed stream 10b, and a small amount of ethyl ether from stream 40b. Stream 45b is fed into a still 500b, where thermal energy is used to

separate all volatile components from water and provide an overhead stream 60b and a bottoms stream 55b. Still 500b is one of only two process elements, in this embodiment of subprocess B, that utilizes thermal energy.

Overhead stream 60b includes ethanol and any trace amount of ether 5 that may have been present in stream 45b. Bottom stream 55b includes water and any other heavy materials. A generally small amount of external heat or energy is needed to provide the separation, due to the different boiling points of water and solvents.

Overhead stream 60b progresses to a condenser 700, where ethanol 10 vapors are condensed to liquid. The resulting liquid stream is fairly pure, typically at least 90% and preferably at least 95%. The ethanol can be collected and used for solvent stream 15b. Bottoms stream 55b is generally sufficiently pure water to allow disposal with a minimum of further purification.

Top exit stream 65b from liquid-liquid extraction unit 400b contains 15 the majority of ether from unit 400b, a major amount of ethanol from stream 30b, and typically includes a small amount of water. Top exit stream 65b and stream 230b are fed into a still 600b, the second of the two process elements of subprocess B in this embodiment that utilizes thermal energy. Top exit stream 65b is separated by still 600b into an overhead stream 75b and a bottoms stream 67.

Overhead stream 75b includes the ether; typically this stream is fairly 20 pure, typically at least 95% pure and preferably at least 98% pure. Overhead stream 75b is recycled into the process and combined with ether stream 80b, out from dryer 300b of subprocess A.

Bottom stream 67 includes the heavier ethanol; this stream is fairly 25 pure, typically at least 90% pure and preferably at least 95% pure. Bottom stream 67, composed of fairly pure ethanol, can be treated in the same manner as stream 60b, either collected, returned to the process as solvent stream 15b, or further purified.

A second alternate embodiment of the process is diagrammatically 30 illustrated in FIG. 9. This process has an initial extraction process that removes the water from the solids followed by two subprocesses. The first subprocess removes the initial solvent from the solids and a second subprocess that separates and

reclaims the water, solvent and other components. The description of this preferred process uses the same reference numerals used before for like streams and equipment, as appropriate, except that the reference numerals are followed by a "c".

FIG. 9 shows a process similar to the process of FIG. 8, except this embodiment includes additional process equipment. Bottom stream 67c from unit 600c is sent to an evaporator unit 900, which is designed to boil off an azeotropic mixture of ethanol and water, to provide stream 76 and stream 77. Stream 76 contains a mixture of ethanol, water preferably and some small amounts of additional volatile material. Stream 76 progresses to system 1000, a series of molecular sieves. System 1000 takes the azeotropic mixture from evaporator unit 900 and provides ethanol, stream 78. Remaining water from the separation leaves system 1000 as stream 79.

The nonvolatilized portion of stream 75c exits unit 900 as stream 77, relatively clean water.

15

A third alternate embodiment of the process is diagrammatically illustrated in FIG. 10. The description of this preferred process uses the same reference numerals used before for like streams and equipment, as appropriate, except that the reference numerals are followed by a "d".

20

FIG. 10 is another embodiment of the process and is similar to the process of FIG. 9. However, the process of FIG. 10 has an added liquid/liquid extractor unit 450. Unlike the embodiments of FIGS. 8 and 9, the aqueous (bottom) stream 45d, from unit 400d does not feed unit 500d directly, but instead is one of two feed streams to unit 450. Similar to unit 400d, unit 450 extracts ethanol from an aqueous feed stream using an ether, which is the other feed stream 46 provided to unit 450. The organic (top) phase stream from unit 450, stream 47 is combined with organic phase stream 65d from unit 400d. Additionally, vapor stream 60d from unit 500d is not sent to a condenser but instead is combined with streams 47 and 65d and the resulting stream is combined with stream 230d from system 200d. This combined stream is fed to unit 600d. Also in this embodiment, stream 67d is split into streams 61 and 62. Stream 62 carries the appropriate amount of ethanol to provide 200-proof ethanol and ethanol to regenerate the sieve beds of system 1000. Stream 61 is sent to a storage tank for reuse in the process.

Alternate embodiments, of any of the process described above, which utilize an initial extraction process that removes the water from the solids followed by two subprocesses, are within the scope of this invention.

5           The various processes described above used two solvents to remove water from solids; specifically, the first solvent replaced the water, and then the second solvent replaced the first solvent. Although the description above labeled solvents as "first solvents" and "second solvents", and the like, it should be recognized that these groupings are not limiting. In some designs, for example, a  
10          solvent listed in the "second solvent" group may be used as a first solvent; similarly, a solvent listed in the "first solvent" group may be used as a second solvent. The only basis is that the second solvent has a heat of vaporization, enthalpy of vaporization, or other such physical property, that is less than that of the first solvent. If a third solvent is used, the third solvent would have a heat of  
15          vaporization, enthalpy of vaporization, or other such physical property, that is less than that of the second solvent.

#### General Operating Conditions

The following generally operating conditions are suitable for the  
20          process according to the invention, when operated in a typical pilot plant scale.

Stream	Flowrate
Feed stream 10	100-120 lbs/min (15-25 gal/min)
First solvent stream 15	Based on stream 10
Solids stream 20	Based on streams 10 and 15, and on stream 70
Second solvent stream 70	Based on stream 20

25          First solvent stream 15 (lb/min)         = about 1.0 to 0.3         (I)  
                  Feed Stream 10 (lb/min)

Second solvent stream 70 (lb/min)         = about 1.1 to 0.3         (II)  
                  Solids stream 20 (lb/min)

30          Process temperature = 85-90 °F  
                Process pressure = atmospheric

The flow rates within the system that are useful in accordance with the invention are indicated above. Generally, feed stream 10 has a flow rate of 100 to 120 lbs/min. The flow rate of first solvent stream 15 is set in accordance with equation (I). The flow rates of second solvent stream 70 generally range from 10-20 lbs/min, but may also be adjusted relative to stream 20 through equation (II). The flow rates of the various streams into and out from subprocess B are generally governed by stream flow rates in system 100 and subprocess A.

#### Exemplary Process Conditions

10                 Provided below are exemplary stream components and proposed material flow rates for a commercial size, modeled process, described in reference to FIG. 7, which used n-propyl bromide as the second solvent. A binary diagram, for n-propyl bromide / ethanol / water is provided as FIG. 11. It was found that using n-propyl bromide, for a system desirous of separating water and ethanol, was  
 15                 beneficial in that there was a tendency for the system to equilibrate at a low water percentage.

	Solids Feed Stream 10a	Ethanol Feed Stream 15a
Flow Rate	4000 lb/min	1090 lb/min
Fiber	12 wt-%	0
Oil	Trace	0
Water	73 wt-%	7.4 wt-%
Glycerol	Trace	0
Acetic Acid	Trace	0
Ethanol	13 wt-%	92.6 wt-%

20                 In unit 100a, feed stream 10a (usually at a temperature of about 85-90 °F) and first solvent stream 15a would be sent through a series of six screen extractor units 116 (see FIG. 2, where a series of three screen extractor units 116A, 116B, 116C are illustrated). The resulting streams from the six extractors would be:

	Flow Rate	Water	Ethanol	Fiber
Extractor #1 solids stream	3543 lb/min	70 wt-%	17 wt-%	11 wt-%
Extractor #2 solids stream	3216 lb/min	65 wt-%	21 wt-%	12 wt-%
Extractor #3 solids stream	2889 lb/min	58 wt-%	27 wt-%	14 wt-%
Extractor #4 solids stream	2562 lb/min	50 wt-%	33 wt-%	15 wt-%
Extractor #5 solids stream	2234 lb/min	40 wt-%	42 wt-%	17 wt-%
Extractor #6 solids stream	1906 lb/min	26 wt-%	53 wt-%	20 wt-%

	Wet Solids Stream 20a	Aqueous Stream 30a
Flow Rate	1906 lb/min	3251 lb/min
Fiber	20 wt-%	3 wt-%
Oil	Trace	0
Water	26 wt-%	78 wt-%
Glycerol	Trace	0
Acetic Acid	Trace	0
Ethanol	53 wt-%	19 wt-%

5 Wet solids stream 20a would progress to unit 200a, where it and second solvent stream 70a would be sent through a series of six screen extractor units 216 (see FIG. 5, where a series of three screen extractor units 216A, 216B, 216C are illustrated). The resulting streams from the six extractors would be:

	Flow Rate	Water	Ethanol	n-PB	Fiber
Extractor #1 solids stream	1799 lb/min	23 wt-%	49 wt-%	6 wt-%	22 wt-%
Extractor #2 solids stream	1691 lb/min	21 wt-%	44 wt-%	12 wt-%	23 wt-%
Extractor #3 solids stream	1583 lb/min	18 wt-%	38 wt-%	20 wt-%	25 wt-%
Extractor #4 solids stream	1475 lb/min	14 wt-%	31 wt-%	29 wt-%	26 wt-%
Extractor #5 solids stream	1368 lb/min	10 wt-%	23 wt-%	38 wt-%	28 wt-%
Extractor #6 solids stream	1260 lb/min	6 wt-%	13 wt-%	50 wt-%	31 wt-%

The miscella stream from Extractor #1 would correspond to stream 230a of FIG. 7. The components of stream 230a are provided below.

The solid stream obtained from Extractor #6 would correspond to stream 5 220a of FIG. 7. Stream 220a, fed into dryer 300a, provides solids stream 90a and vapor stream 80a. Solids stream 90a would be a flow of 389 lb/min of 100% solids

	Vapor stream 80a	Miscella stream 230a	Combined Stream 80a/230a
Flow Rate	871 lb/min	1410 lb/min	2281 lb/min
Water	8 wt-%	30 wt-%	21 wt-%
Ethanol	19 wt-%	62 wt-%	46 wt-%
n-PB	72 wt-%	7 wt-%	32 wt-%

Combined stream 80a/230a would be fed into the top of separation 10 column 400a and aqueous stream 30a would be fed into the bottom of column 400a and streams 45a and 65a would exit. In this example, no additional solvent, as stream 40a, was added.

	Top Organic Stream 45a	Aqueous Bottom Stream 65a
Flow Rate	655 lb/min	4877 lb/min
Water	1 wt-%	61 wt-%
Ethanol	2 wt-%	34 wt-%
n-PB	93 wt-%	3 wt-%
Oils	4 wt-%	0 wt-%
Fiber/solids	0 wt-%	2 wt-%

15 Stream 45a would be fed to still 500a and the exiting streams 60a, 55a would have the compositions listed below. In this example, a steam sparge stream, at 35 lb/min, was added to carry or otherwise facilitate transporting the solvents to the top of the still. Stream 65a would be fed to still 600a and the exiting streams 75a, 50a would have the compositions listed below. In this example, heat

exchangers would be used for flashing steam 65a prior to entering still 600a; this would decrease the entering mass flow rate to about 4700 lb/min.

	Vapor Stream 60a	Oil Recovery Stream 55a	Ethanol Recovery 75a	Water Stream 50a
Flow Rate	665 lb/min	25 lb/min	1706 lb/min	3003 lb/min
Water	6 wt-%	0.6 wt-%	6 wt-%	95 wt-%
Ethanol	2 wt-%	0 wt-%	92 wt-%	0 wt-%
n-PB	92 wt-%	0 wt-%	2 wt-%	0 wt-%
Oils/glycerine	0 wt-%	99.3 wt-%	0 wt-%	2 wt-%
Fiber/solids	0 wt-%	0 wt-%	0 wt-%	3 wt-%

#### Additional Exemplary Process Conditions

- Provided below are exemplary stream components and proposed material flow rates for a modeled process described in reference to FIG. 10, which used ether as the second solvent.

	Solids Feed Stream 10d		Ethanol Feed Stream 15d	
Component	wt-%	lb/min	wt-%	lb/min
Fiber	12.2	398.2	0	0
Oil	0.6	19.6	0	0
Water	69.9	2281.5	4.5	88.1
Glycerol	1.2	39.2	0	0
Acetic Acid	0.1	3.3	0	0
Ethanol	16.0	522.2	82.8	1621.6
Ether	0	0	12.7	248.7
Total	100	3264	100	1958.4

	Wet Solids Stream 20d		Aqueous Stream 30d	
Component	wt-%	lb/min	wt-%	lb/min
Fiber	44.1	394.2	0.1	4.0
Oil	1.1	9.8	0.2	9.8
Water	10.6	94.8	52.6	2274.9
Glycerol	2.2	19.6	0.5	19.6
Acetic Acid	0	0	0.1	3.2
Ethanol	36.5	326.4	42.0	1817.4
Ether	5.6	50.1	4.6	198.7
Total	100	894.9	100	4327.5

	Ether Feed Stream 40d		Ether Feed Stream 70d	
Component	wt-%	lb/min	wt-%	lb/min
Fiber	0	0	0	0
Oil	0	0	0	0
Water	0	0	0	0
Glycerol	0	0	0	0
Acetic Acid	0	0	0	0
Ethanol	3.0	120.5	3.0	26.8
Ether	97.0	3894.7	97.0	868.1
Total	100	4015.2	100	894.9

	Wet Solids Stream 220d		Liquid Stream 230d	
Component	wt-%	lb/min	wt-%	lb/min
Fiber	46.9	390.3	0.4	3.9
Oil	0.0	0.2	1.0	9.6
Water	5.7	47.4	4.9	47.4
Glycerol	0.0	0.4	2.0	19.2
Acetic Acid	0.0	0.0	0.0	0.0
Ethanol	0.4	3.3	36.5	350.0
Ether	46.9	390.3	55.1	527.8
Total	100	831.8	100	958.0

	Dried Solids Stream 90d		Ether Solvent Stream 80d	
Component	wt-%	lb/min	wt-%	lb/min
Fiber	89.0	390.3	0	0
Oil	0	0.2	0	0
Water	10.8	47.4	0	0
Glycerol	0.1	0.4	0	0
Acetic Acid	0	0	0	0
Ethanol	0	0	0.8	3.3
Ether	0	0.4	99.2	389.9
Total	100	438.7	100	393.2

	Bottoms Stream 45d		Top Stream 65d	
Component	wt-%	lb/min	wt-%	lb/min
Fiber	0	0	0.1	4.0
Oil	0	0	0.2	9.8
Water	79.5	2274.9	0	0
Glycerol	0.7	19.6	0	0
Acetic Acid	0.1	3.2	0	0
Ethanol	18.4	527.0	25.7	1410.8
Ether	1.2	35.1	74.0	4058.3
Total	100	2859.8	100	5482.9

	Ethanol Stream 60d		Water Stream 55d	
Component	wt-%	lb/min	wt-%	lb/min
Fiber	0	0	0	0
Oil	0	0	0	0
Water	5.0	27.7	99.0	2247.1
Glycerol	0	0	0.9	19.6
Acetic Acid	0	0	0.1	3.2
Ethanol	95.0	527.0	0	0
Ether	0.0	0.0	0	0
Total	100	554.8	100	2270.0

Component	Ether Product Stream 75d		Ethanol Stream 67d	
	wt-%	lb/min	wt-%	lb/min
Fiber	0	0	0.4	7.9
Oil	0	0	1.1	19.4
Water	0.1	2.4	2.5	40.0
Glycerol	0	0	1.1	19.2
Acetic Acid	0	0	0	0
Ethanol	1.0	46.3	94.9	1714.5
Ether	99.0	4585.7	0	0.5
Total	100	4634.5	100	1806.4

Only three pieces of the process equipment from the system depicted in and described with reference to FIG. 10 use thermal energy. Dryer 1300, which is a Schnecken tube-type dryer, uses an exemplary 77.3 lb/min of steam, still 1700  
 5 uses an exemplary 6532 lb/min of steam, and water separator 1500 uses an exemplary 199.5 lb/min of steam.

The above specifications provide a complete description of the process, equipment, and compositions of the invention. Since many embodiments of  
 10 the invention can be made without departing from the spirit and scope of the invention, the invention resides in the claims hereinafter appended.

**WE CLAIM:**

1. A process for drying solids initially wet with water, the process comprising:
  - (a) contacting a feed stream comprising solids having interstitial spaces, and water present in the interstitial spaces with a first solvent;
  - (b) displacing the water present in the interstitial spaces with the first solvent to provide solids with the first solvent in the interstitial spaces;
  - (c) contacting the feed stream having the first solvent in the interstitial spaces with a second solvent; and
  - (d) displacing the first solvent present in the interstitial spaces with the second solvent to provide solids having the second solvent in the interstitial spaces.
2. The process according to claim 1, further comprising the step of:
  - (e) removing the second solvent from the solids by the application of heat.
3. The process according to claim 1, wherein the step of contacting a feed stream with a first solvent comprises:
  - (a) contacting a feed stream with a first solvent having a heat of vaporization lower than the heat of vaporization of water and is soluble with water
4. The process according to claim 3, wherein the step of contacting the feed stream with a second solvent comprises:
  - (a) contacting the feed stream with a second solvent having a heat of vaporization lower than the heat of vaporization of the first solvent.
5. The process according to claim 4, wherein the step of contacting the feed stream with a second solvent comprises:

(a) contacting the feed stream with a second solvent that is miscible with the first solvent.

6. The process according to claim 5, wherein the step of contacting a feed stream with a first solvent comprises:

(a) contacting a feed stream with a first solvent that is an alcohol.

7. The process according to claim 6, wherein the step of contacting a feed stream with a first solvent that is an alcohol comprises:

(a) contacting a feed stream with a first solvent that is ethanol.

8. The process according to claim 5, wherein the step of contacting the feed stream with a second solvent comprises:

(a) contacting the feed stream with a second solvent that is n-propyl bromide.

9. The process according to claim 5, wherein the step of contacting the feed stream with a second solvent comprises:

(a) contacting the feed stream with a second solvent that is an ether.

10. The process according to claim 9, wherein the step of contacting the feed stream with a second solvent that is an ether comprises:

(a) contacting the feed stream with a second solvent that is one of ETBE and MTBE.

11. The process according to claim 1, wherein:

(a) contacting a feed stream with a first solvent comprises contacting a feed stream with an alcohol;

(b) contacting the feed stream with a second solvent comprises contacting the feed stream with n-propyl bromide; and the process further comprises:

(c) obtaining an ether product that is at least 95% pure ether; and

(d) obtaining an alcohol product that is at least 90% pure alcohol.

12. The process according to claim 11, wherein the step of obtaining an alcohol product that is at least 90% pure ethanol comprises:

(a) obtaining an alcohol product that is at least 95% pure ethanol.

13. The process according to claim 1, wherein:

(a) contacting a feed stream with a first solvent comprises contacting a feed stream with an alcohol;

(b) contacting the feed stream with a second solvent comprises contacting the feed stream with an ether; and the process further comprises:

(c) obtaining an ether product that is at least 95% pure ether; and

(d) obtaining an alcohol product that is at least 90% pure ethanol.

14. The process according to claim 13, wherein the step of obtaining an alcohol product that is at least 90% pure ethanol comprises:

(a) obtaining an alcohol product that is at least 95% pure ethanol.

15. A process for drying solids initially wet with water, the process comprising:

(a) providing a feed stream comprising solids having interstitial spaces, and water present in the interstitial spaces;

(b) providing an ethanol source stream;

(c) providing an ether source stream;

(d) displacing the water present in the interstitial spaces with the ethanol to provide solids with ethanol in the interstitial spaces;

(e) displacing the ethanol present in the interstitial spaces with n-propyl bromide to provide solids with n-propyl bromide in the interstitial spaces;

(f) removing the n-propyl bromide from the solids by the application of heat.

16. The process according to claim 15, wherein said step of displacing the water present in the interstitial spaces with the ethanol to provide solids with ethanol in the interstitial spaces comprises:

- (a) in a solid-liquid extractor, displacing the water present in the interstitial spaces with the ethanol to provide solids with ethanol in the interstitial spaces; and
- (b) obtaining an aqueous stream comprising the water and ethanol.

17. The process according to claim 16, further comprising:

- (a) feeding the aqueous stream to a liquid-liquid extractor;
- (b) providing a second ether source stream; and
- (c) processing the aqueous stream and the second ether source stream in the liquid-liquid extractor to obtain an aqueous bottom stream comprising water and ethanol and a top organic stream comprising ether and ethanol.

18. The process according to claim 17, further comprising:

- (a) distilling the aqueous bottom stream to obtain an ethanol stream and a water stream.

19. The process according to claim 15, wherein said step of displacing the ethanol present in the interstitial spaces with n-propyl bromide to provide solids with n-propyl bromide in the interstitial spaces comprises:

- (a) in a second solid-liquid extractor, displacing the ethanol present in the interstitial spaces with ether to provide solids with ether in the interstitial spaces.

20. A process for drying solids initially wet with water, the process comprising:

- (a) providing a feed stream comprising solids having interstitial spaces, and water present in the interstitial spaces;
- (b) providing a first solvent from a source stream;
- (c) providing a second solvent from a source stream;

- (d) displacing the water present in the interstitial spaces with the first solvent to provide solids with the first solvent in the interstitial spaces;
- (e) displacing the first solvent present in the interstitial spaces with the second solvent to provide solids the second solvent in the interstitial spaces; and
- (f) removing the second solvent from the solids by the application of heat.

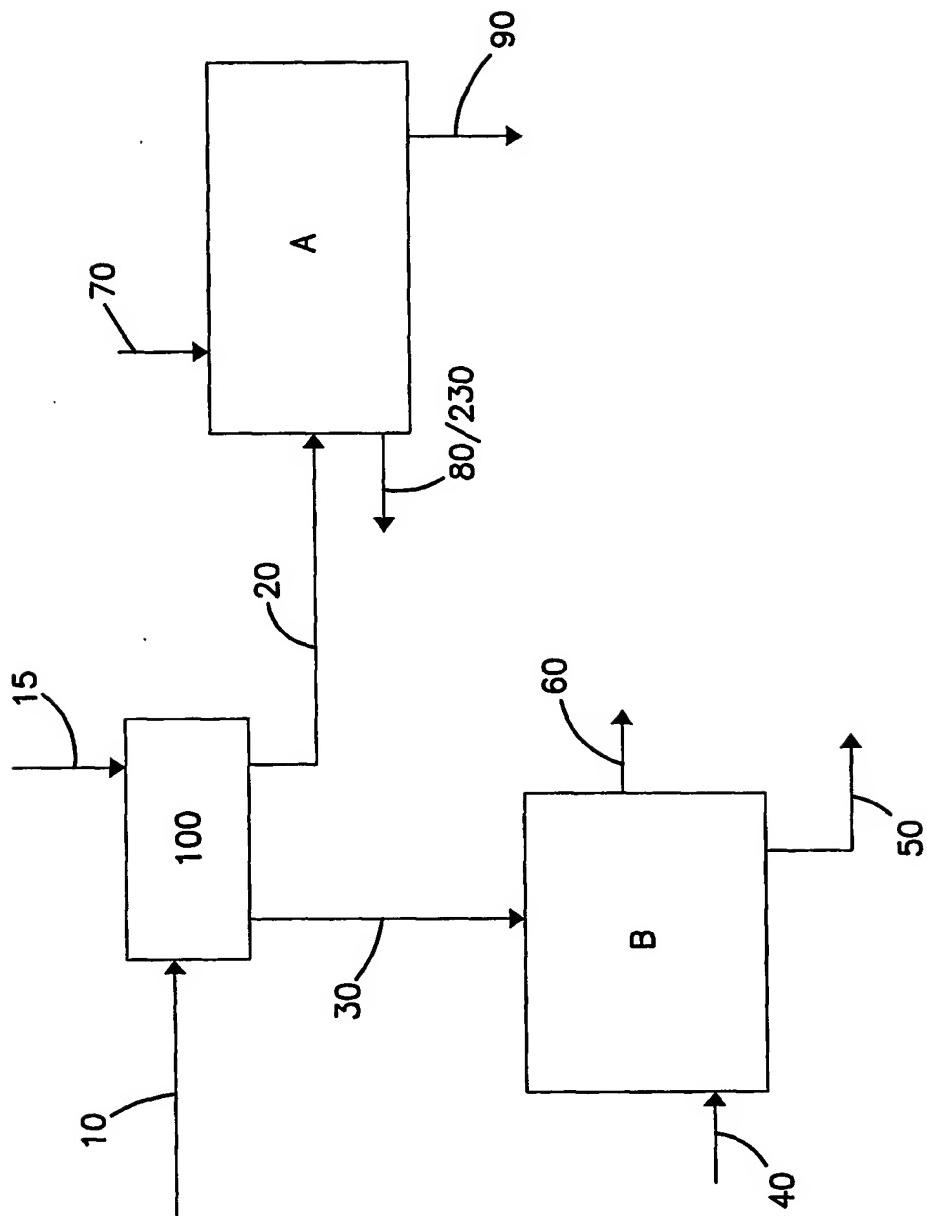
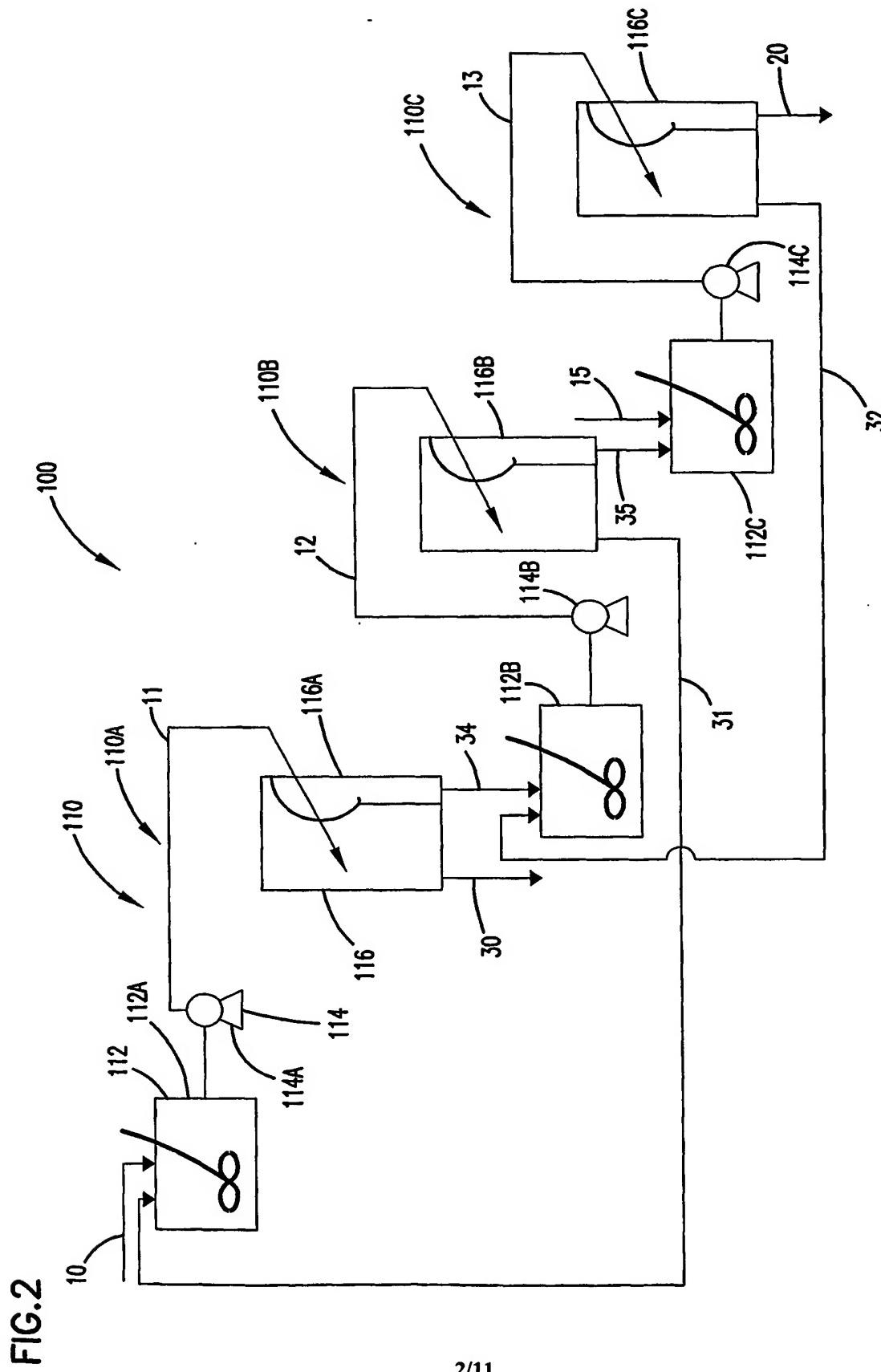


FIG. 1



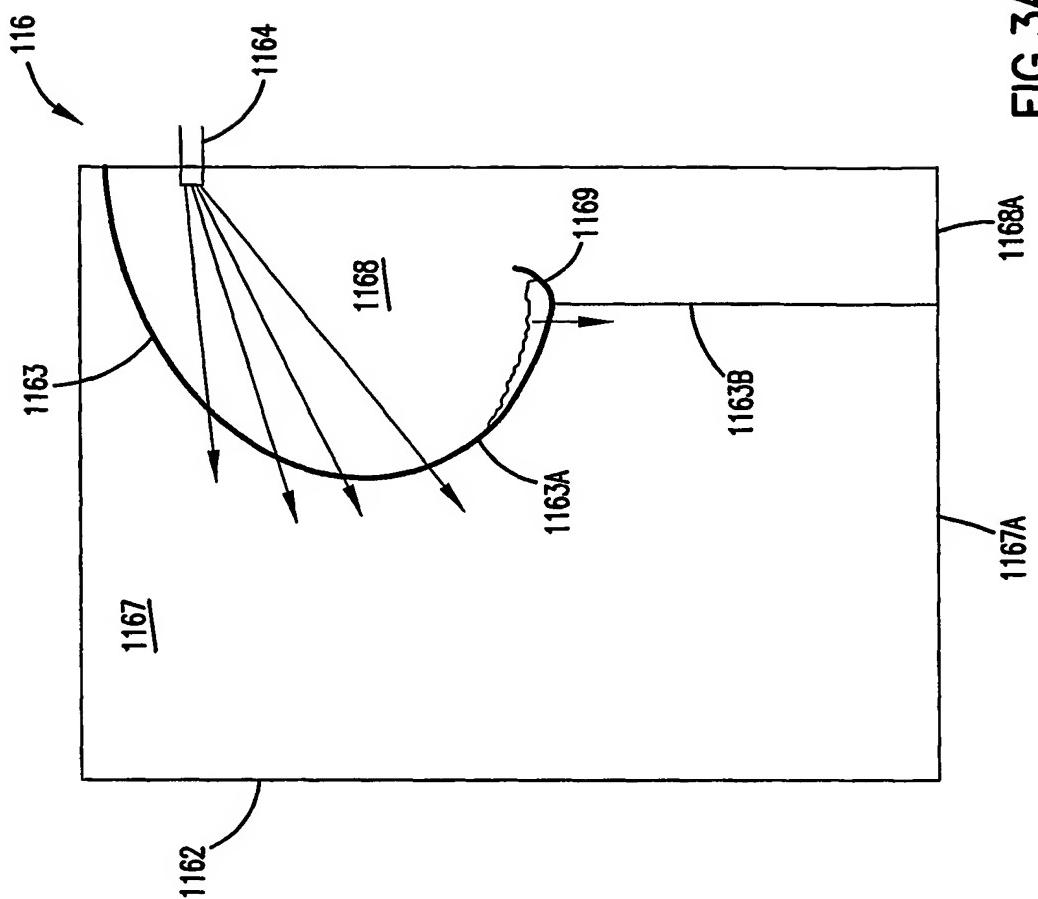


FIG. 3A

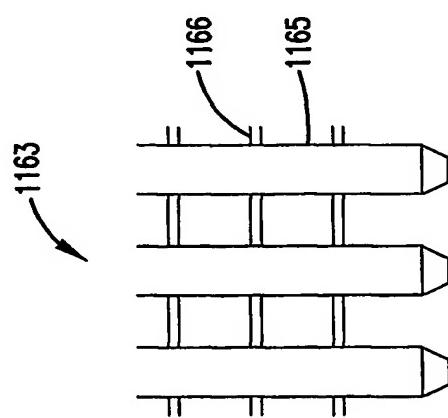


FIG. 3B

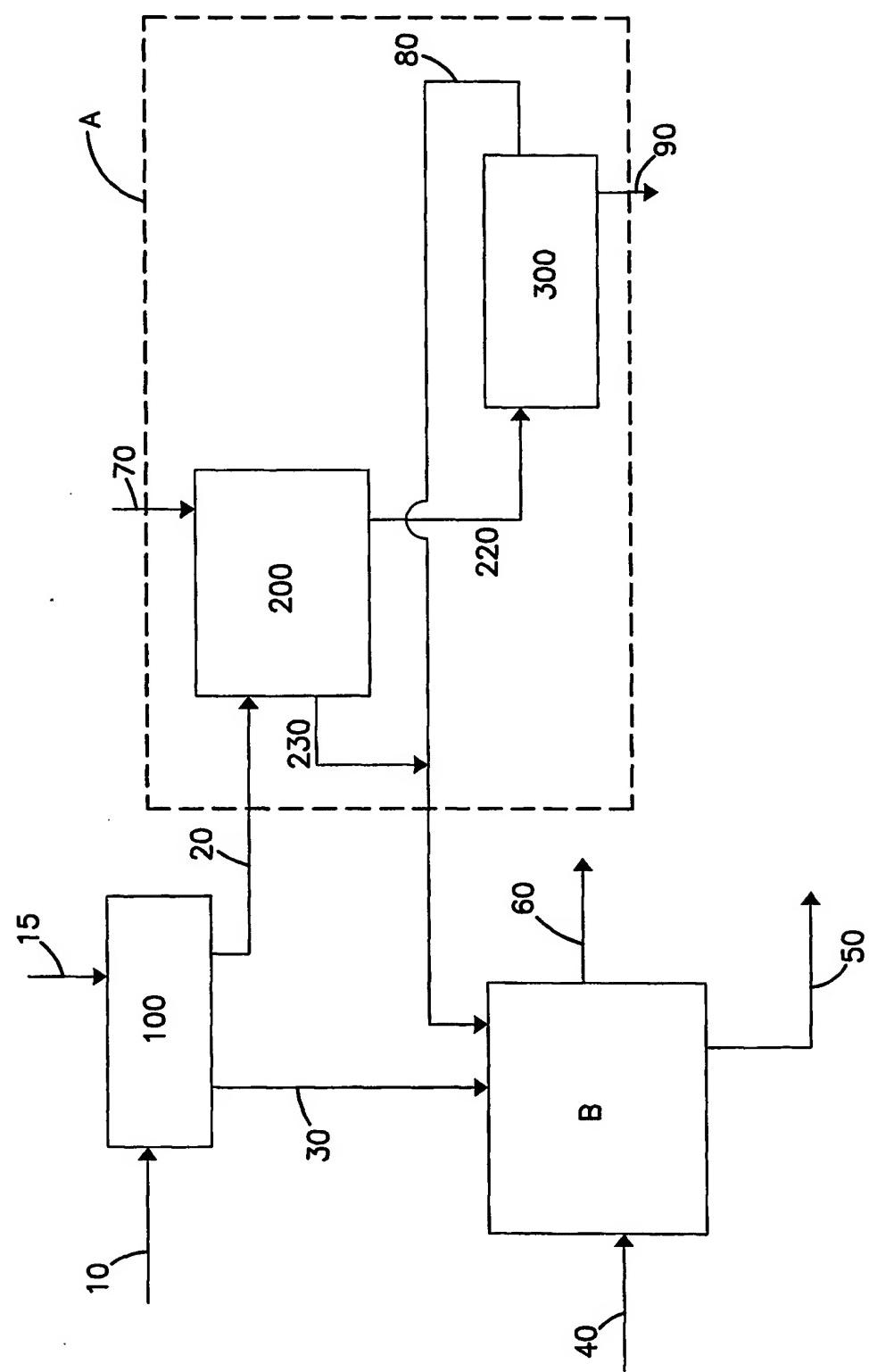
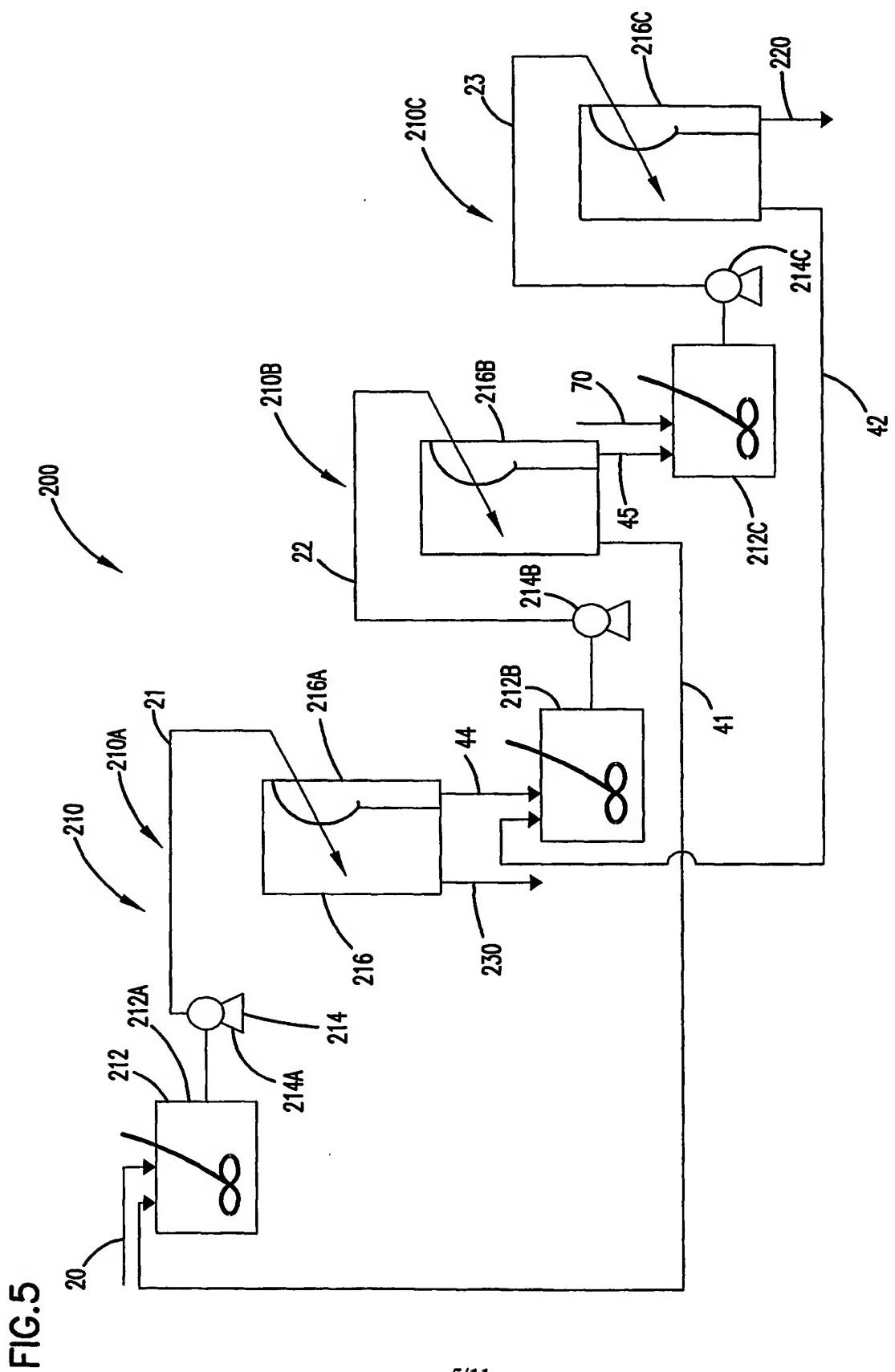


FIG.4



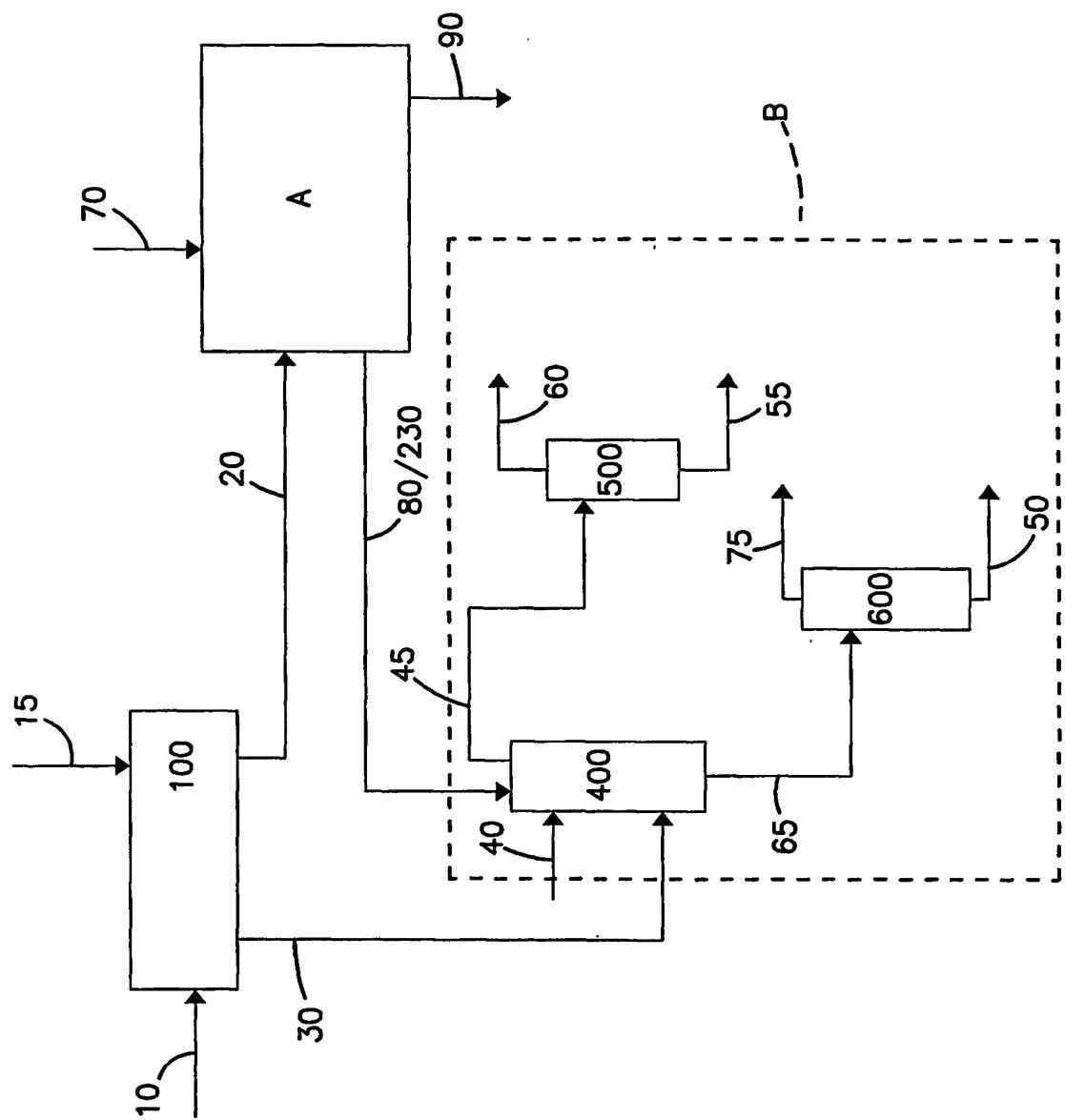


FIG.6

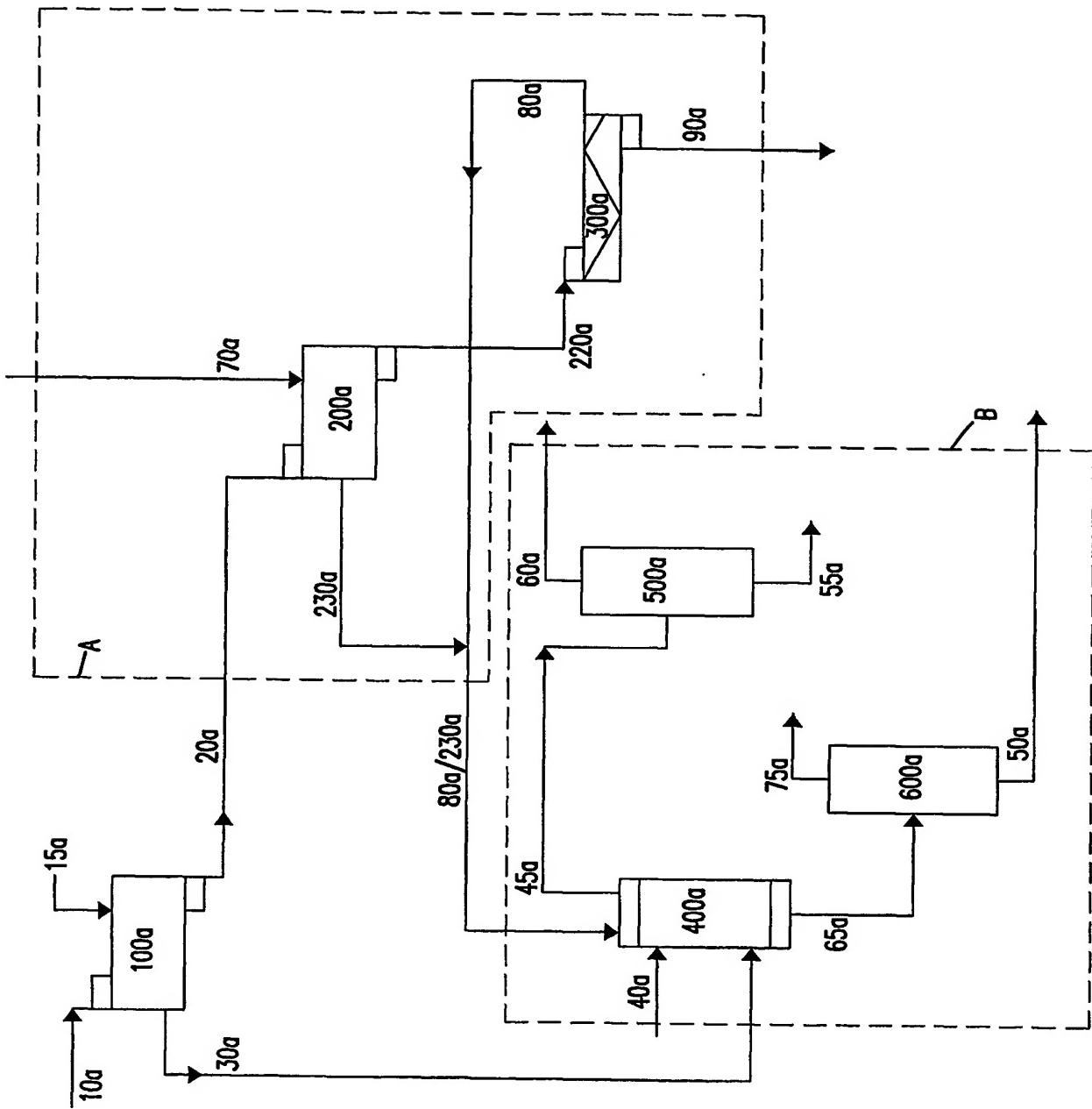


FIG. 7

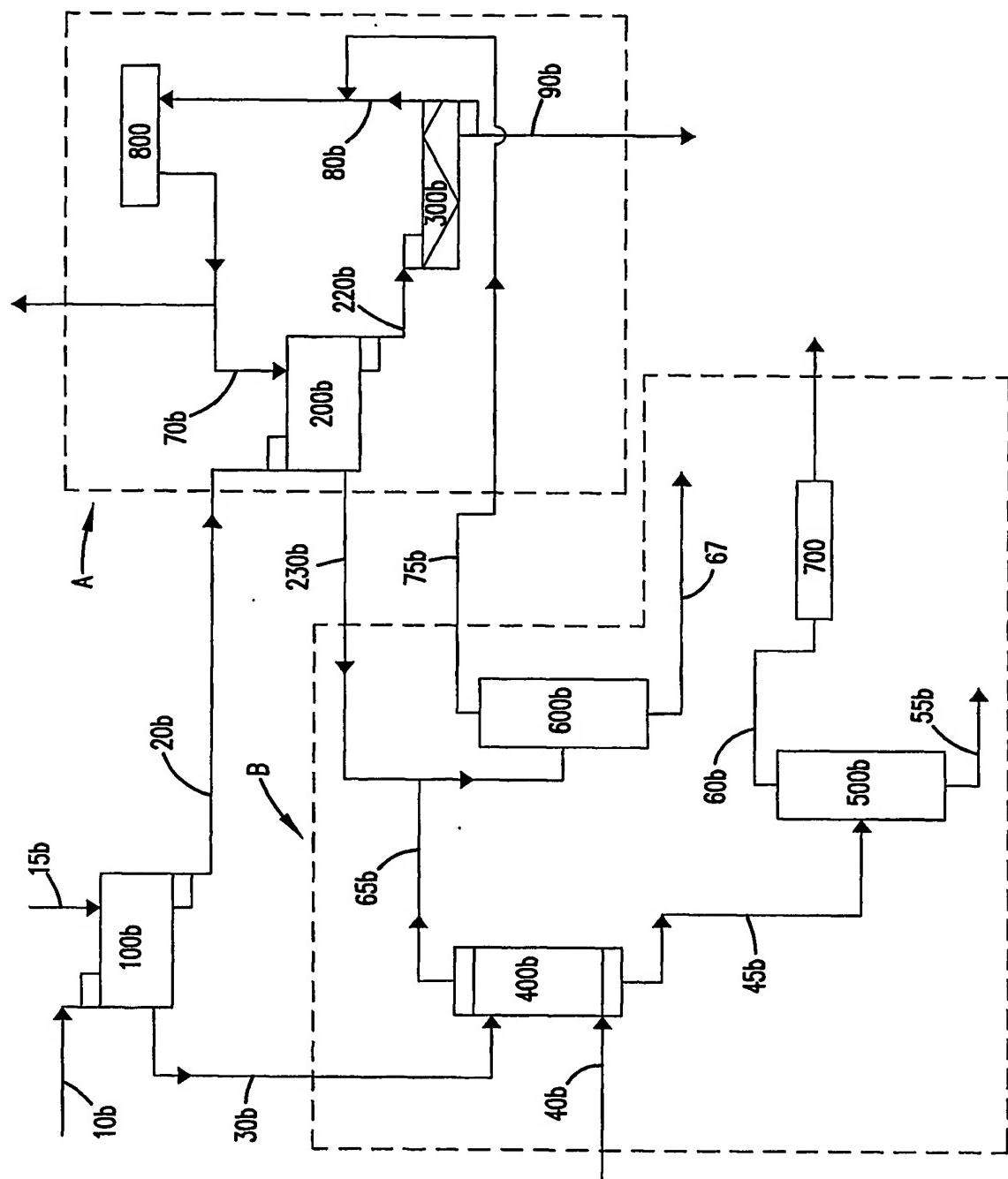


FIG. 8

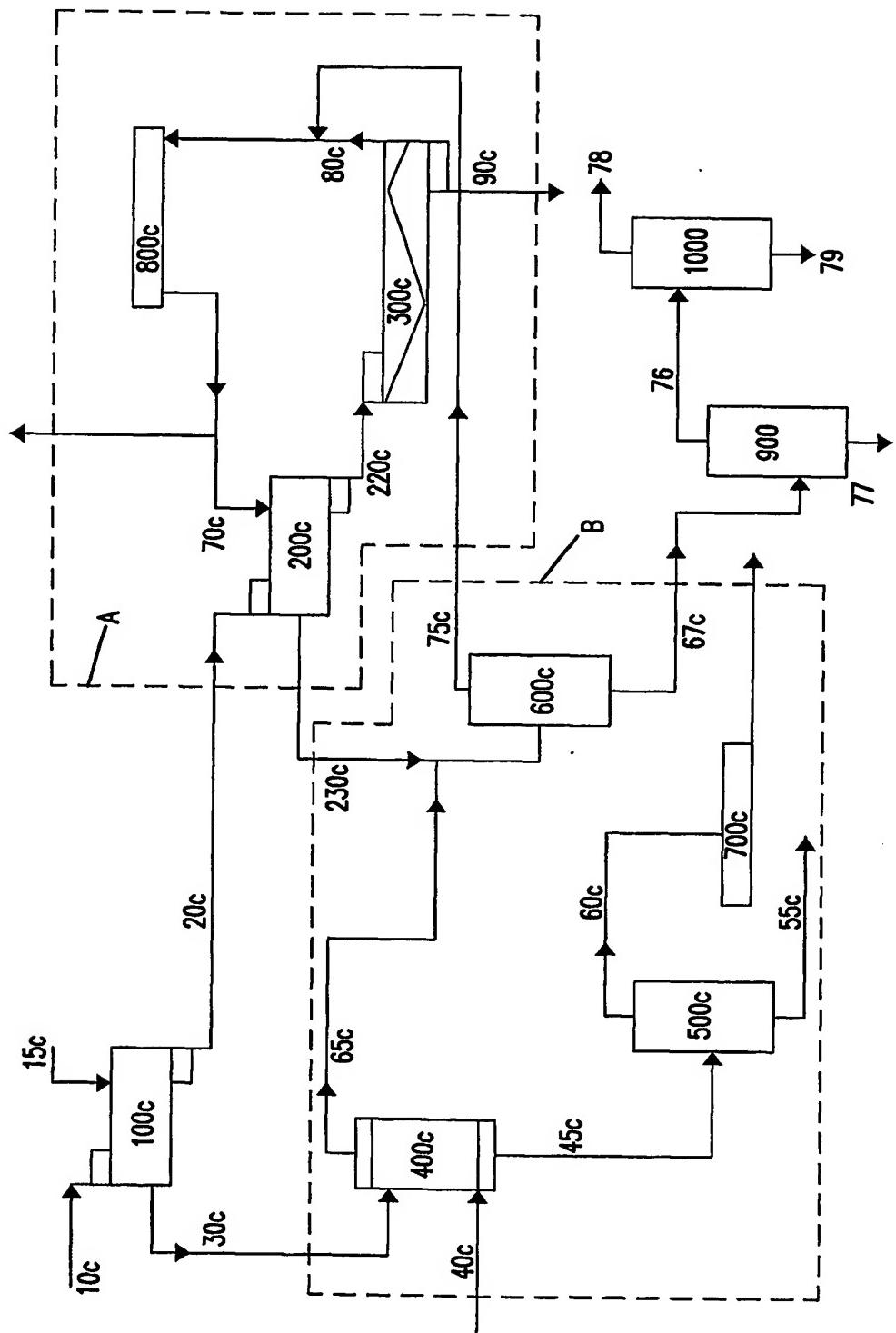


FIG.9

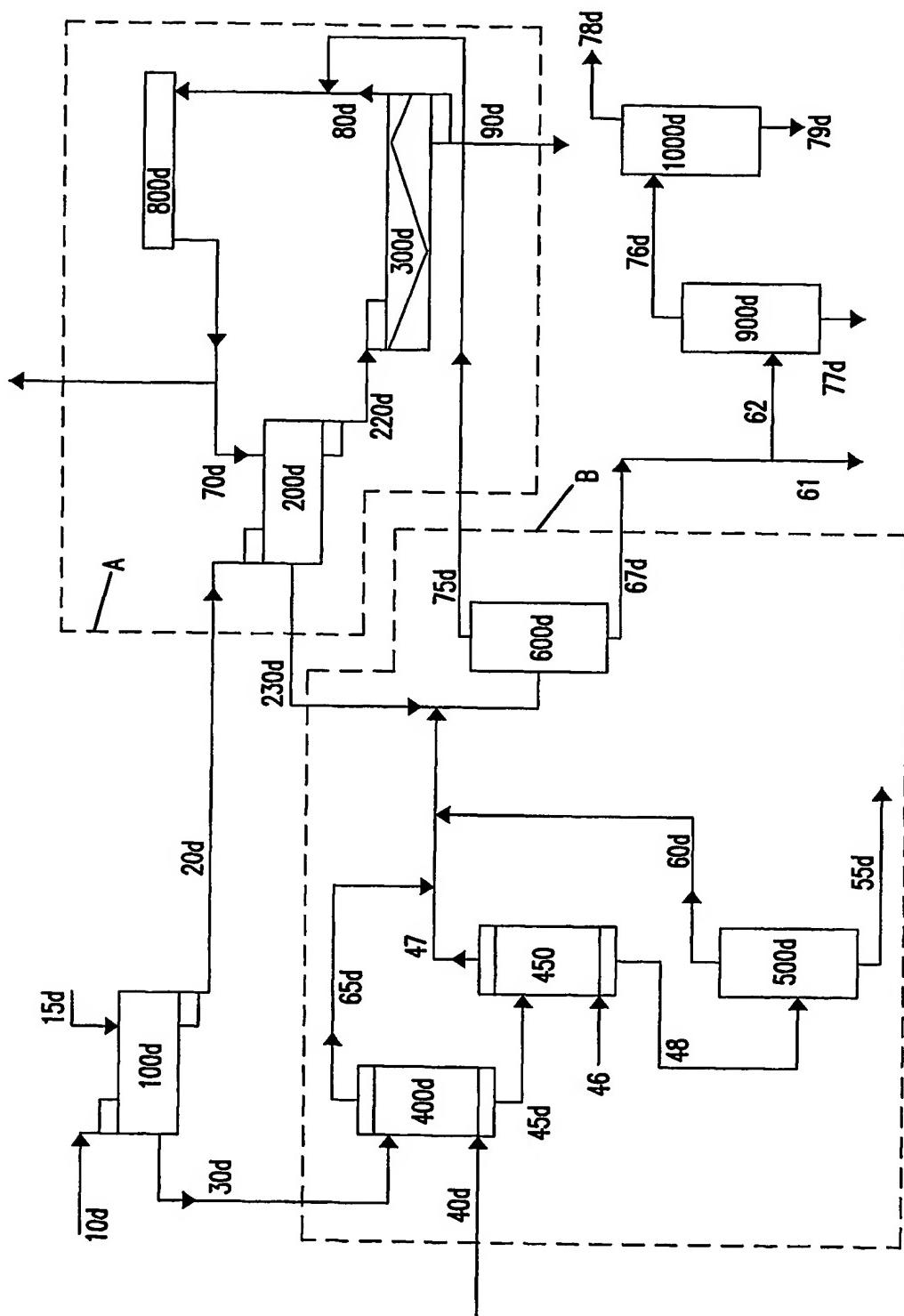


FIG.10

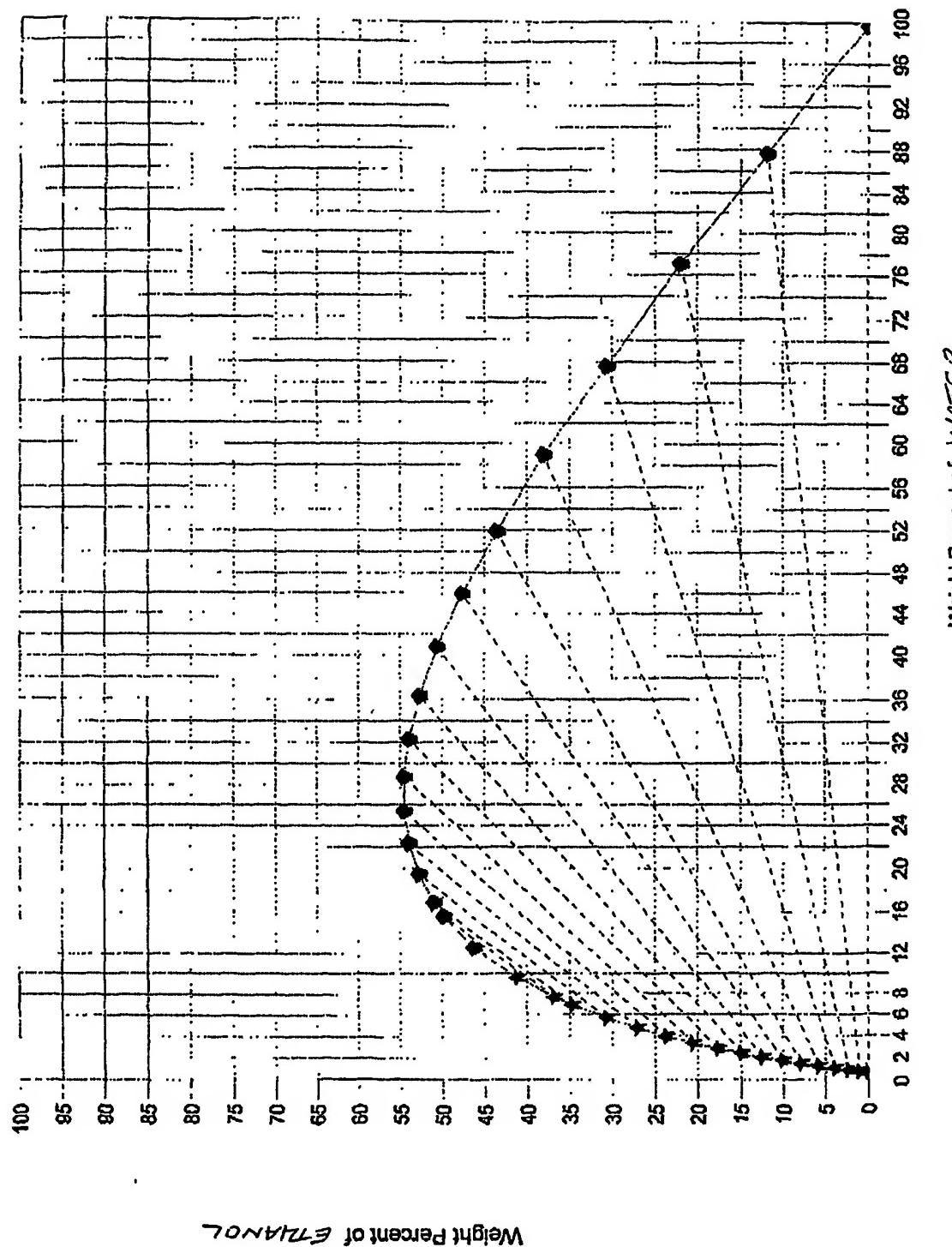


FIG. 11

Weight Percent of ETHEROL

# INTERNATIONAL SEARCH REPORT

International application No.

PCT/US03/40646

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) : F26B 3/00  
US CL : 34/340

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
U.S. : 34/340,337,342; 210/634,639,770,774; 426/11,425,429,430

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X,P ---	US 6,516,537 A (TEICH et al) 11 February 2003, see especially column 6, line 46-column 7, line 41.	1,2,20
Y,P		3-10,15 and 16
X ---	US 6,438,867 A (TEICH et al) 27 August 2000, see especially column 6, lines 15-44 and column 7, lines 13-44.	1,2,20
Y		3-10,15 and 16
Y,P	US 6,017,505 (ZEIGLER et al) 25 January 2003, see especially column 4, line 43-column 5, line 5.	3-10

Further documents are listed in the continuation of Box C.

See patent family annex.

Special categories of cited documents:	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A" document defining the general state of the art which is not considered to be of particular relevance	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E" earlier application or patent published on or after the international filing date	"Y"	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&"	document member of the same patent family
"O" document referring to an oral disclosure, use, exhibition or other means		
"P" document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search

22 April 2004 (22.04.2004)

Date of mailing of the international search report

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